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# TRANSMITTANCE OF THIN FILMS IN THE EXTREME ULTRAVIOLET®

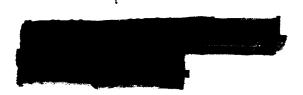
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### SUMMARY

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The extreme ultraviolet transmittance of thin unbacked films of Parlodion,  $Aluminum, and Indium was measured over the wavelength range extending to ~150~{\rm \AA}\,.$ 

The use of Aluminum for eliminating stray light in solar spectroscopy from rockets is illustrated with solar spectra and spectroheliograms made at wavelengths near 300 Å.



<sup>\*</sup>Supported, in part, by National Aeronautics and Space Administration Contract

#### INTRODUCTION

In extreme ultraviolet research, thin films are very useful for several reasons. First, they may be used as windows, transmitting the radiation but holding back a gas, for example, to isolate gas-filled detectors from the vacuum system; second, their selective transmission properties make them useful as optical filters for eliminating stray light or for order-sorting.

Many years ago celluloid in the form of a thin film was shown by Laird (1,6) to transmit to wavelengths as short as 450 Å, and also in the soft x-ray range. Later, quantitative measurements of the transmittance of celluloid in the range 1000 to 300 Å were made by O'Bryan (2), and Tomboulian and Bedo (3) measured the transmittance of zapon from 260 Å to 80 Å.

The discovery by Wood (4) in 1933 that the alkali metals transmit ultraviolet but no visible radiation, supplied the impetus for investigations concerning the use of thin metal films as optical filters. Because of their chemical activity, however, the alkali metals can only be used if sealed, in sandwich fashion, between quartz plates, as has been accomplished by O'Bryan (5). The quartz, of course, restricts their use to wavelengths longer than about 1550 Å. Gold and silver have long been known to transmit in restricted wavelength ranges, gold at 5000 Å and silver in a narrow band near 3200 Å. Although silver was reported by Laird (6) to transmit wavelengths as short as 900 Å, no use has been made of its transmitting properties in the vacuum ultraviolet as yet.

The first quantitative work on the transmittance of thin unbacked metal films in the extreme ultraviolet was done by Tomboulian and Pell (7), who measured aluminum over the wavelength range 80 to 320 Å, and by Astoin and Vodar (8) for aluminum

supported on collodion over the wavelength range 750 to 130 Å. This spectral region includes the L<sub>1</sub> and L<sub>2,3</sub> x-ray edges, of which only the latter was clearly observed. More recently, Walker, Rustgi, and Weissler (9) have measured the transmittance of a number of thin metal films in the extreme ultraviolet. Hunter (10) has determined the extinction coefficient and index of refraction for aluminum and indium over most of the spectral range where they are transparent, thus providing information from which to calculate the transmittance of films of a given thickness, including all the interference effects. In the present paper, the results of transmittance measurements for aluminum, indium, and parlodion films will be presented, and their possible use as filters in the extreme ultraviolet will be discussed.

## **PARLODION**

Parlodion films were prepared by the conventional method of placing a drop of a dilute solution of paroldion in amyl acetate on a distilled water surface. The films were quite uniform, and thin enough to be black by reflected light. Parlodion is a trade name for cellulose nitrate, and the composition is  $C_{12}H_{16}$  (NO<sub>3</sub>)<sub>4</sub>. Celluloid, or collodion, is cellulose nitrate with some camphor added.

The transmittance of a film of parlodium approximately 270 Å thick, as measured interferometrically, is shown in Fig. 1. Shown also for comparison is the transmittance of a celluloid film approximately 100 Å thick, measured by O'Bryan (2).

The curves for parlodion and celluloid are very similar. Both show an absorption maximum near 800 Å. For this reason their usefulness as windows and substrates for other metallic films is limited to wavelengths below about 500 Å or above approximately 1000 Å. If the data for celluloid are recomputed for 270 Å thickness,

the curve lies fairly close to the curve for parlodion. There seems to be a deeper minimum for parlodion, and there are several other minima indicating absorption bands which are considered to be real. It is not clear from the data of O'Bryan whether additional minima would have been present had measurements been made at a larger number of wavelengths.

#### **ALUMINUM**

Thin unbacked films of aluminum free from pinholes were prepared in the following fashion. A glass substrate was coated in vacuum with fuchsin as a parting agent, and then coated with aluminum under optimum conditions as described by Hass, Hunter, and Tousey (11). It was important to keep the entire vacuum chamber very clean and to avoid the use of shutters of a type that might scatter dust particles when opened or closed. Before coating with fuchsin the glass was cleaned by removing in vacuum a thin collodion film that had been placed on its surface prior to introduction into the vacuum chamber. After coating, the aluminum film was floated off the substrate and onto a water surface from which it was picked up on a thin stainless steel plate containing a 6 mm diameter hole, which it covered. For making larger unbacked films, it was necessary to mount them on fine mesh screens. An 80 per inch mesh screen of 80% transmittance was quite satisfactory. Aluminum filters 15 × 20 mm in area, having a thickness of the order of 1000 Å could be prepared in this way without pinholes. A detailed description of the process will be published by Angel (12).

Previous work on aluminum transmission has been carried out, either on films deposited on a plastic substrate such as zapon (7) or collodion (8) or with aluminum deposited under conditions such that its reflectance was rather low (9). The earlier

results showed clearly that transparency commences shortward of the characteristic wavelength, 837 Å, corresponding to the electron eigenloss, reaches a high value just longward of the  $L_{2,3}$  absorption edge 170 Å, and then falls to a low value on the short wavelength side of the edge, and rises again below about 120 Å. The different measurements were not in close agreement, and it appeared probable that the transmittance depended on the conditions of preparation to a considerable extent.

Transmittance data obtained on an 800 Å thick aluminum film evaporated under the best of conditions and with no backing, are shown in Fig. 2. This is approximately the minimum thickness required to be completely opaque for visible and near ultraviolet. For wavelengths longer than 476 Å the data are in good agreement with the results of Walker, Rustgi, and Weissler (9), if account is taken of the different thicknesses of the films. From 300 to 150 Å, the data agree well with the results of Tomboulian and Pell (7). However, the structure reported by them on the long wavelength side of the absorption edge was not found and may be associated with the fact that they deposited the aluminum on a zapon substrate, as they suggest.

The wavy appearance in the transmittance curve with minima near 500 Å, 580 Å, and 660 Å, was also present in the data of Walker, Rustgi, and Weissler (9). This can be explained as an interference effect, produced by the presence of an oxide layer on the aluminum surface. The transmittance of an 800 Å thick aluminum film with no oxide layer was calculated from the optical constants obtained by Hunter (10), and is shown in Fig. 2. A second curve was calculated assuming an oxide layer of 40 Å thickness on both sides of the aluminum film and using approximate optical constants for aluminum oxide determined from reflectance measurements for a synthetic sapphire surface down to 584 Å, and extrapolated to 480 Å. Interference

effects were included in the calculation and it appears that they do, indeed, produce minima qualitatively similar to those observed in the actual aluminum film.

It is obvious from Fig. 2 that a large fraction of the absorption is produced by the oxide layer. If it were possible to produce an aluminum film without an oxide layer, the transparency would remain at a high level from 170 Å, at least to 500 Å and perhaps much closer to the critical wavelength. Thicker films will transmit nearly as well as an 800 Å film, for  $\lambda > 170$  Å. To shorter wavelengths, however, it is important to use the thinnest possible film.

Aluminum film filters are extremely useful in spectroscopy. Figure 3 shows the spectrum of a spark source obtained by Tilford (13) with a 3-meter Hilger grazing incidence spectrograph, and two exposures with a 1000 Å thick aluminum film introduced between the slit and the grating. The spectrum, longward of 170 Å, is photographed with little reduction in intensity. Many lines can be seen shortward of the  $L_{2,3}$  edge to 150 Å, somewhat reduced in intensity. Other spectra, not reproduced here, show some transmittance from 150 to 45 Å, but with strong absorption bands centered at 129 Å and 95 Å.

The aluminum film filter is especially useful in extreme ultraviolet photographic solar spectroscopic work from rockets. Because the extreme ultraviolet portion of the sun's radiation is only of the order of  $10^{-6}$  of the total radiation, stray visible and near ultraviolet light produces an extremely high background when using diffraction grating spectrographs. By interposing an unbacked aluminum filter, which is completely opaque at wavelengths longward of 837 Å, it is possible to eliminate entirely the stray light, which would otherwise swamp the spectrum.

Two types of photograph which could not have been made without the aluminum filter are shown in Fig. 4. They were obtained from an Aerobee-Hi rocket by the Naval Research Laboratory on May 10, 1963 (14). In the center is a spectrum obtained with a small grazing-incidence spectrograph employing a 40-cm radius, 600 line/mm diffraction grating at 85° incidence. It can be seen that the spectrum is completely free from stray light background. Above and below are monochromatic images of the sun in the form of spectra. These were obtained with a simple concave grating, used at normal incidence, and receiving directly the parallel light from the sun. The grating was of 40 cm radius of curvature and 600 lines/mm. A thin aluminum film supported on a wire mesh was placed about 1 cm in front of the photographic film. The dispersion in printing was adjusted so as to match the grazing incidence spectrum. The most intense solar image is from the He II. 304 Å resonance line. Superimposed upon the sun's surface can be seen six active regions. Shown for comparison are CaK and H-alpha spectroheliograms taken from the ground on the same day. The second most prominent solar image at 257 Å is from a blend of lines, including Si X from the corona. Between these two images it is possible to see a third image produced by the Fe XV line at 284 Å. This shows only in the active regions and is produced by the sun's corona. Also visible at 335 Å are the same plage regions in the emission from the Fe XVI line.

## INDIUM

Unbacked films of indium have been investigated by Walker, Rustgi, and Weissler (9), who showed that a film 800 Å thick begins to transmit shortward of 1100 Å, reaches a maximum transmittance of 17% at 770 Å, then drops suddenly to 0.3% at 730 Å.

The long wavelength transmittance limit is close to the critical wavelength determined by the electron eigenloss.

Attempts were made to produce unbacked indium films in the same manner as aluminum, by evaporating onto fuchsin, or sodium iodide as parting agents. It was impossible, however, to remove the indium film by immersion in water. Sugaraerosol, however, as used by Curcio and Carpenter (15) proved to be satisfactory as a parting agent. Evaporation of indium took place from a tungsten boat under excellent vacuum conditions and was completed in a few seconds. The transmittance curves for two indium films are shown in Fig. 5. It can be seen that indium films are excellent filters for transmitting the range 1000 to 750 Å. The transmittance of the films shown in Fig. 5 is considerably greater over the entire wavelength range than the values reported by Walker, Rustgi, and Weissler (9) for a film approximately one-half as thick.

Unlike aluminum, for indium there appears to be little or no absorption by a surface layer. This is apparent from the agreement with the curves shown by the dotted lines, which were calculated assuming no absorbing surface layer and employing the optical constants determined from reflection measurements by Hunter (10). As a result, there is no loss of transmittance with age. Interference effects produced by internal reflections were observed in the thinner of the measured films, but for the thicker they were too small to detect. It is possible, however, that a surface layer of some kind is present, because the undulations in the measured curve are greater than in the calculated curve. This might be produced by a surface layer of modified refractive index, but negligible absorption.

As an example of the use of indium film filters, the spectrum of a spark source obtained (14) with the Hilger grazing incidence 3-meter spectrograph is shown in Fig. 6.

In Fig. 7 is a similar exposure for the wavelength range 150 to 49 Å. It is clear that indium transmits not only from 1000 to 750 Å, but also from 120 Å to shorter wavelengths, probably as far as the M-1 edge at 15 Å. Transmittance values at 44.3 and 67 Å were measured by Blake, Meekins, and Unzicker (16) to be 19.5 and 37 per cent, respectively.

### SUMMARY

Figure 8 is a diagram showing transparency ranges for a number of metals and semi-conducting materials, most of which can be prepared in the form of unbacked films. This has been assembled from a variety of sources and is intended as a stimulus for further work. The arrowheads indicate the positions of the critical wavelengths, from the electron eigenloss values tabulated by Pines (17). Dotted lines indicate expected trends not as yet verified by experiment. The positions of the various x-ray edges are introduced together with observed or supposed ranges of transparency on the long wavelength side.

It is a pleasure to thank D. W. Angel for preparing the films of aluminum and indium, and G. Fritz for the parlodion films.

## REFERENCES

- 1. Laird, E. R., Phys. Rev. 29, 41 (1927).
- 2. O'Bryan, H. M., J. Opt Soc. Am. 22, 739 (1932).
- 3. Tomboulian, D. H, and Bedo, D. E., Rev. Sci. Instr. 26, 747 (1955).
- 4. Wood, R. W., Nature 131, 582 (1933); Phys. Rev. 44, 353 (1933).
- 5. O'Bryan, H. M., Rev. Sci. Instr. 6, 328 (1935).
- 6. Laird, E. R., Phys. Rev. <u>15</u>, 543 (1920).
- 7. Tomboulian, D. H., and Pell, E. M., Phys. Rev. 83, 1196 (1951).
- 8. Astoin, N., and Vodar, B., J. Phys. et Rad. 14, 424 (1953).
- Walker, W. C., Rustgi, O. P., and Weissler, G. L., J. Opt. Soc. Am. 49, 471 (1959).
- 10. Hunter, W. R., Proceedings of this Colloquium.
- 11. Hass, G., Hunter, W. R., and Tousey, R., J. Opt. Soc. Am. 46, 1009 (1956).
- 12. Angel, D. W., Naval Research Laboratory (to be published).
- 13. We are indebted to Dr. S. G. Tilford of NRL for these spectra.
- 14. We are indebted to J. D. Purcell, W. E. Austin and D. Garrett of NRL for these spectra.
- 15. Carpenter, F. E., and Curcio, J. A., Rev. Sci. Instr. 21, 675 (1950).
- 16. Blake, R. L., Meekins, J. F., and Unzicker, A. E. (private communication).
- 17. Pines, D., Rev. Mod. Phys. 28, 184 (1956).

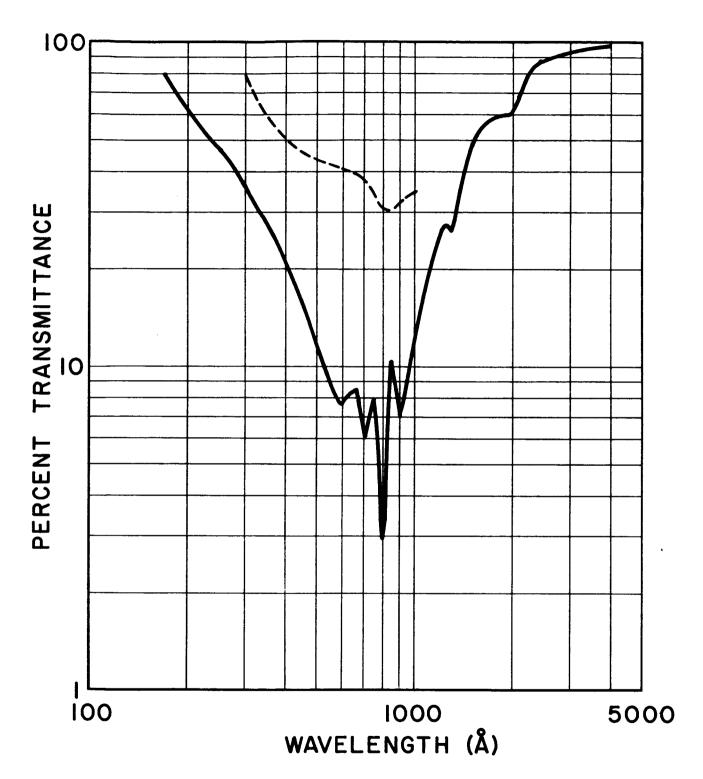
# LIST OF CAPTIONS

- Figure 1. The transmittance of a parlodion (nitrocellulose) film 270 Å thick, (solid curve); and of a celluloid film 100 Å thick as measured by O'Bryan (2).
- Figure 2. The transmittance of an unbacked film of aluminum, 800 Å thick.

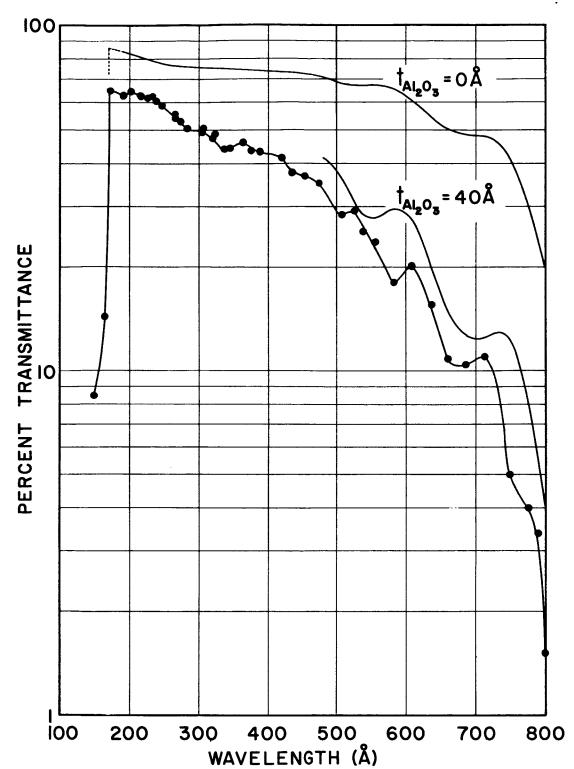
  Included for comparison are curves for aluminum calculated from the optical constance, for the cases of no oxide, and 40 Å oxide on both surfaces.
- Figure 3. The spectrum of a vacuum spark, exposed 1 min. with no filter, and 1 min. and 1.5 min. with an aluminum filter; (S. G. Tilford, U. S. Naval Research Laboratory).
- Figure 4. Solar spectra photographed by Austin, Garrett, Purcell, and Tousey of the Naval Research Laboratory from an Aerobee Hi rocket on May 10, 1963, with instruments making use of a thin aluminum filter to eliminate stray light. The top and bottom spectra were made with a normal incidence concave grating spectrograph receiving parallel light from the sun, and producing a series of monochromatic solar images.

- Figure 5. The spectrum of a vacuum spark exposed 0.5 min. with no filter and 10 min. through an indium film approximately 2000 Å thick (S. G. Tilford, U. S. Naval Research Laboratory.
- Figure 6. The spectrum of a vacuum spark exposed 1.2 min. with no filter and 6 min. through an indium filter approximately 2000 Å thick (S. G. Tilford, U. S. Naval Research Laboratory).
- Figure 7. The measured transmittance of indium films, and curves calculated from the optical constants.
- Figure 8. A summary diagram of the transmission properties of several materials, most of which can be prepared as thin unbacked films.

  X-ray edges are shown as steps; the arrows show critical wavelengths derived from values of the electron eigenloss; dotted sections indicate ranges where there are no data.



TRANSMITTANCE OF A PARLODION FILM APPROX. 270A THICK.



TRANSMITTANCE THROUGH AN UNBACKED AT FILM APPROX. 800Å THICK. CALCULATED TRANS. WITH O & 40Å OF OXIDE ON BOTH SIDES OF THE FILM SHOWN FOR COMPARISON.

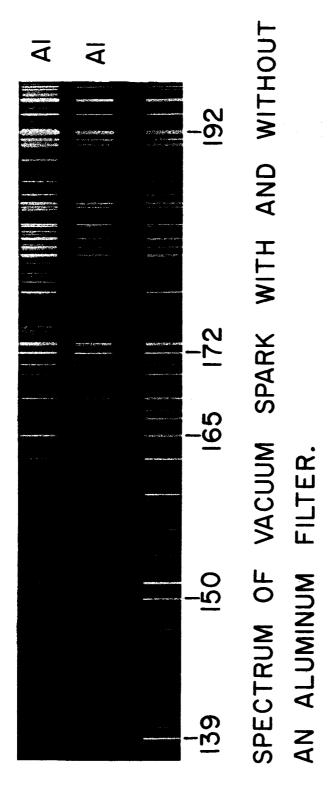


Figure 3

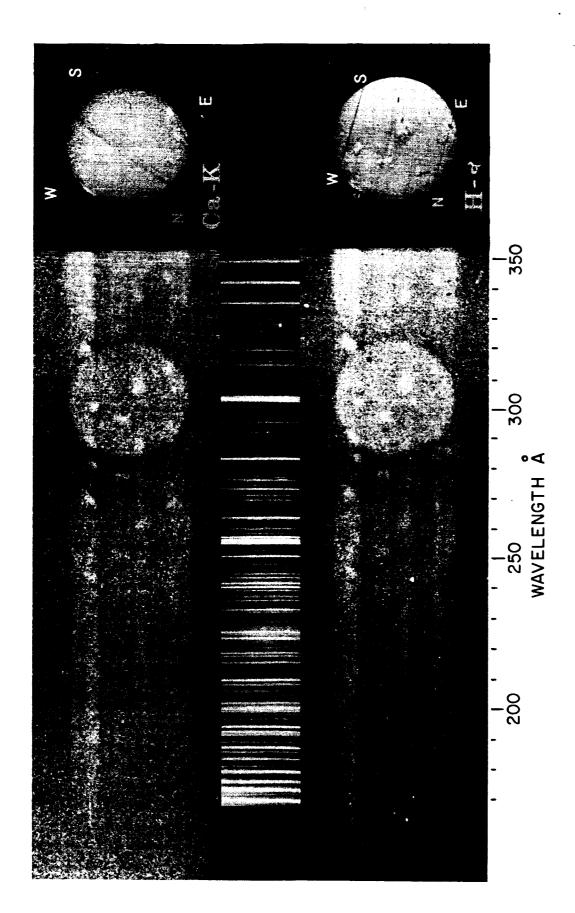


Figure 4

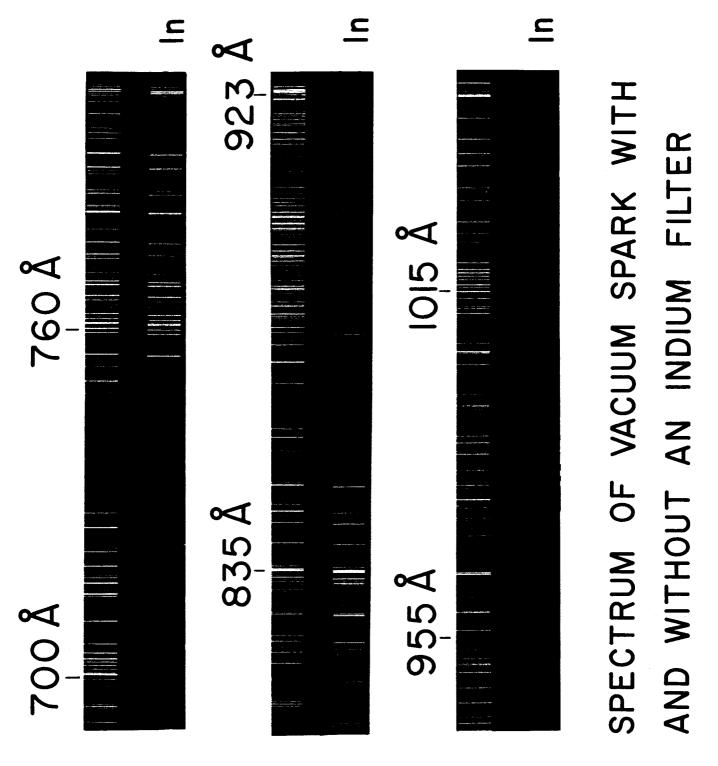
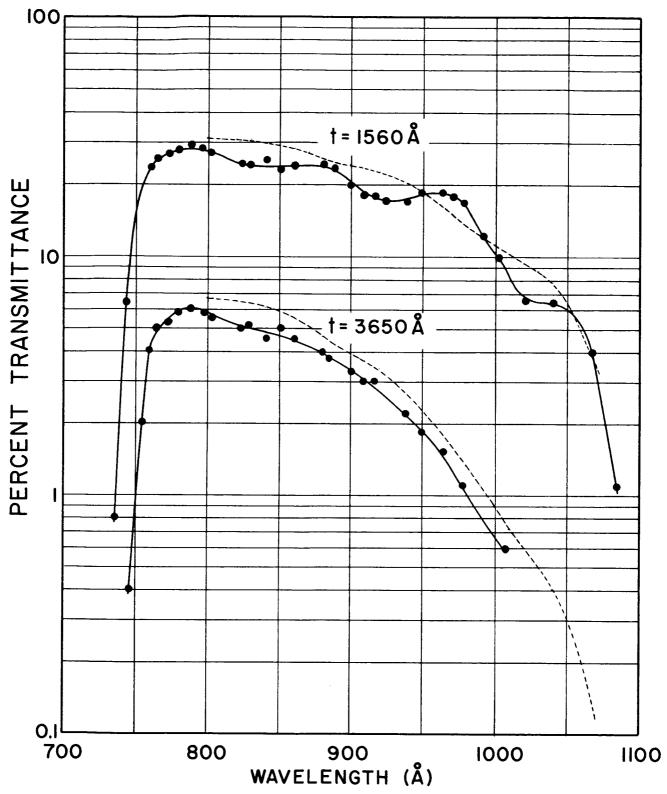


Figure 5



Figure 6



TRANSMITTANCE OF INDIUM FILMS. DASHED LINES ARE CALCULATED VALUES.

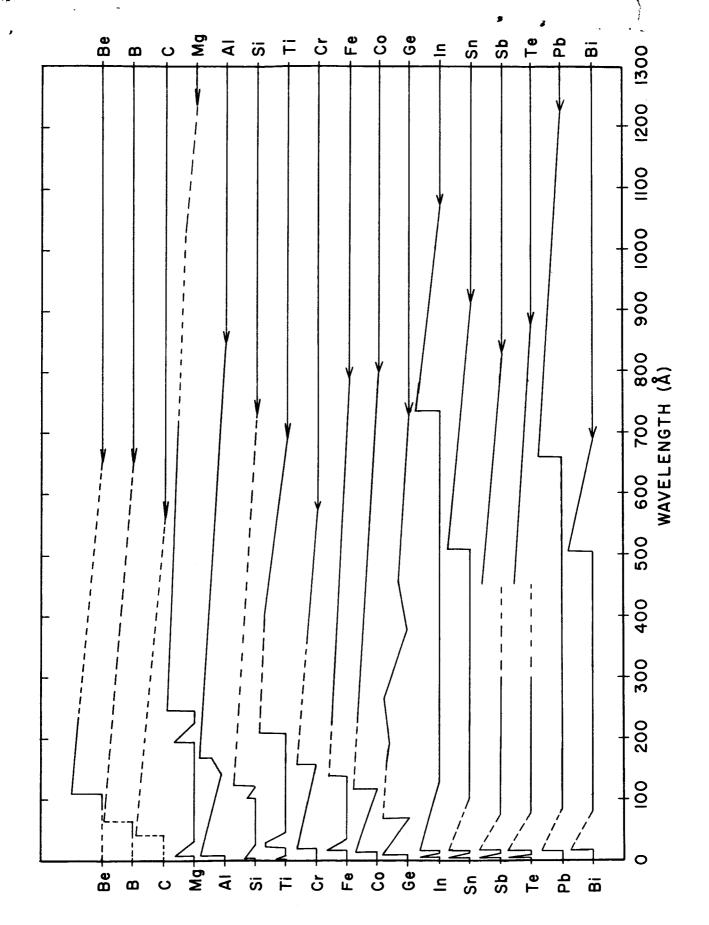


Figure 8